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DMIC review OF RECENT DEVELOPMENTS
Nickel-and Cobalt-Base Alloys
H. J. Wagner • October 27, 1967

ALLOY DEVELOPMENT

NASA Turbine Bucket Program

A final report on the development of nickel-base alloys for jet-engine buckets has been issued by TRW.[1] It describes a cast alloy, VI A, with stress-rupture properties representing a 50 degree F improvement over present-day superalloys; its ductility, while below average, does not suffer adversely in the 1400 to 1600 F range, nor is it prone to the formation of embrittling phases such as sigma, Laves, or mu. The composition of the alloy is as follows:

C	Cr	Mo	Ti	Al	Co	W	Re	Hf
0.13	6.1	2	1	5.4	7.5	5.8	0.5	0.43

Zr	B	Ta	Cb	Ni
0.13	0.02	9	0.5	Bal

Figure 1 shows the stress-rupture properties for some of the experimental cast alloys and compares them with commercial alloys.

Four wrought alloys were also said to show promise in comparison with Udimet 700, and further property evaluations were suggested to determine their full potential.

Air Force Wheel/Bucket Alloys

Universal-Cyclops has completed its evaluation of three wrought nickel-base all ys for advanced wheel/bucket applications: René 85, Unitemp AF 2-1D, and Unitemp AF 2-1DA. On the basis of all the properties investigated, AF 2-1DA was selected for scale-up to production size.[2] This alloy had better workability, strength, and stability than the other two and showed no tendency for sigma formation. Figure 2 is a Larson-Miller parameter plot for the three alloys.

NASA Vane Alloys

TRW has completed a program for NASA-Lewis to develop a cobalt-base alloy for stator vanes in advanced gas-turbine engines.[3] In all, 85 alloys were evaluated in eight different series. Initially, 15 elements were employed as alloy additions,

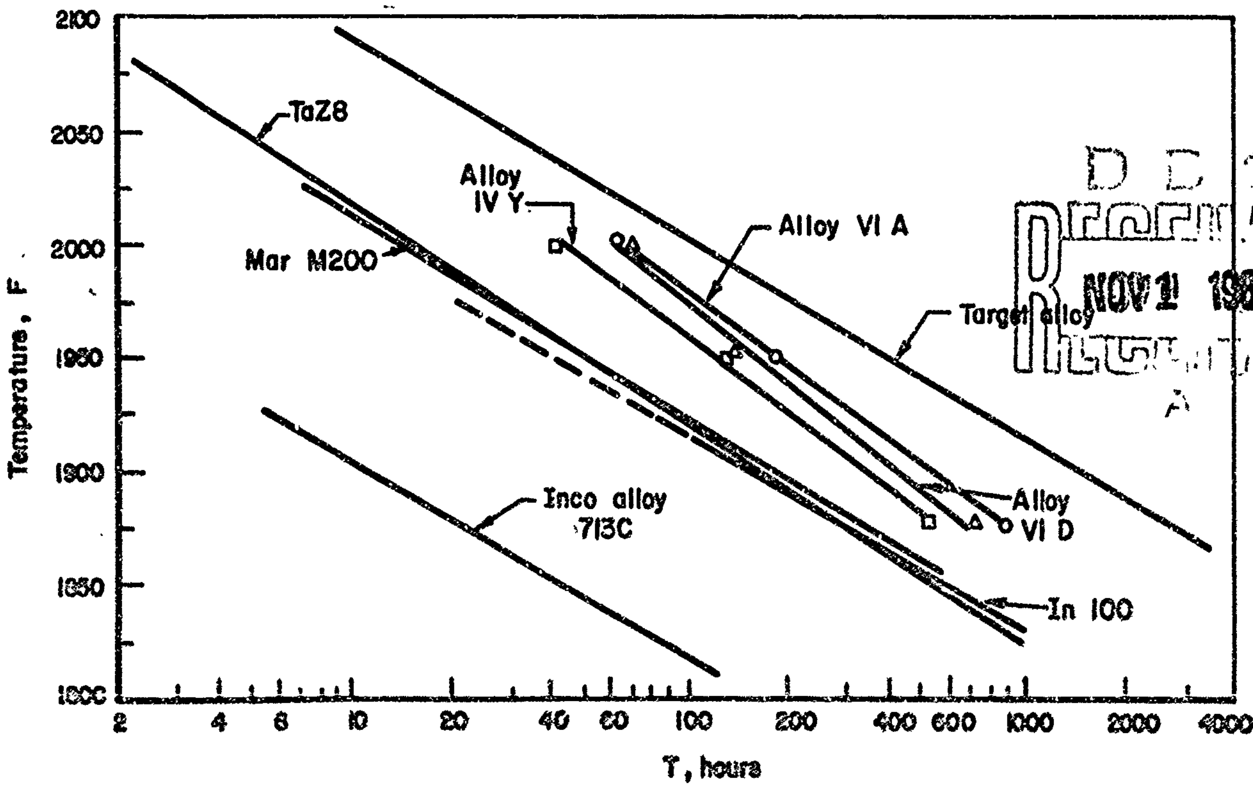

FIGURE 1. CREEP-RUPTURE PROPERTIES AT 15,000 PSI STRESS OF THREE EXPERIMENTAL CAST NICKEL-BASE ALLOYS DEVELOPED BY TRW UNDER A NASA CONTRACT(1)

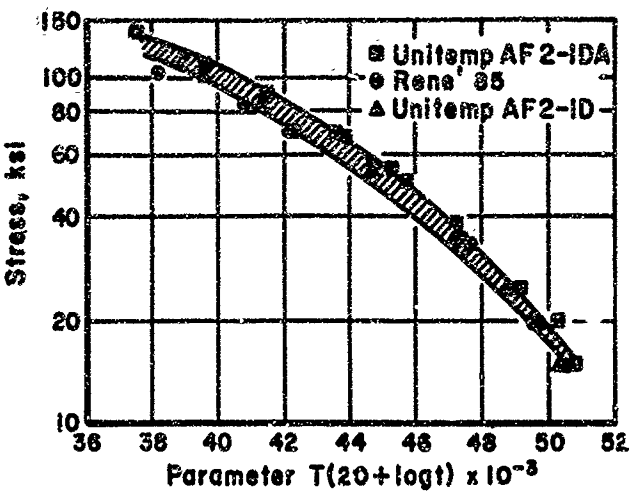

FIGURE 2. LARSON-MILLER PARAMETER CURVES COMPARING RENÉ 85, UNITEMP AF 2-1D, AND UNITEMP AF 2-1DA[2]

namely, tungsten, tantalum, carbon, zirconium, titanium, chromium, nickel, vanadium, boron, hafnium, molybdenum, columbium, manganese, rhenium, and ruthenium. However, during the program, the alloying elements that proved most effective were tungsten, tantalum, carbon, zirconium, titanium, chromium, nickel, and vanadium.

The best alloy developed exhibited a mean creep-rupture life of 24 hours at 8000 psi at 2125 F under an argon atmosphere. The target composition of this alloy in weight percent was 19W, 11Ta, 1.5C, 0.85Zr, 1.1Ti, 7.0Cr, 9.0Ni, 1.7V, and balance Co. While the alloy represents an improvement over commercial cobalt-base alloys, it did not exhibit sufficient strength to indicate that the program goal of a 3000-hour life at 2125 F and 4000 psi stress (in an argon atmosphere) would have been achieved. The alloy that was considered best in terms of creep-rupture life appeared to have been strengthened by a combination of (Ta,Zr,Ti)C precipitates and solid-solutioning effects. It was also found that the best alloy was rolled at 2125 F without any cracking. However, alloys with higher carbon contents had a laminar constituent in their microstructure that prevented them from rolling well.

Air Force Hot Compressor Alloy

General Electric has rolled the five experimental alloys (see Nickel- and Cobalt-Base Alloys Review, August 11, 1967) intended for use as a disk in the aft stages of the compressor in advanced turbojet engines.[4] The alloys were heat treated and evaluated for tensile properties at room temperature to 1400 F and for creep-rupture at 1200 F. The tensile data are shown in Figure 3 and the creep-rupture results are shown in Figure 4. Also plotted for comparison are the best data from the earlier X 1400 program and an internal GE goal curve for an alloy termed René 70XD. Alloy 3 exhibited the highest yield strength and best strength retention with increased temperature. However, instead of the normal solution treatment of 2000 F followed by a 1400 F aging treatment, additional heat treatment suggested further improvement of the low-temperature properties of the alloys. For example, an additional aging at 1200 F, either before

or after aging at 1400 F, was considered. Additionally, cold working of the alloys in combination with heat treatment was a possibility that was suggested.

MICROSTRUCTURAL INSTABILITY

Phases in Nickel-Base Superalloys

In their study of instability in nickel-base superalloys, TRW found that Unitemp AF 2-1DA contained only MC and $M_{23}C_6$ carbides after a "sigma age" (1500 hours at 1600 F).[5] X-ray results showed no trace of sigma or mu in either this alloy or in its companion, AF 2-1D. It was considered that the latter is a borderline case similar to Udimet 700, in which small variations in heat chemistry will determine whether it is a stable or unstable alloy. However, AF 2-1DA was considered to be a "safe" alloy.

Five nickel-base superalloys were studied to determine the type and the temperature of precipitation of minor phases and the gamma-prime solution temperature. The results of this examination indicated that complete solution of gamma prime did not occur in the cast alloys, IN-100 and B-1900, even at the highest exposure temperatures studied (2200 F). Complete solution of gamma prime occurred as follows:

Wrought Alloy	Temperature Range, F
U700	2000 to 2100
Unitemp AF 2-1D	2000 to 2100
René 41	1900 to 2000.

Minor phases were found as follows:

Alloy	Constituent
IN-100	MC, $M_{23}C_6$, M_3B_2
U700	MC, $M_{23}C_6$, M_3B_2
René 41	MC, $M_{23}C_6$, M_6C, mu
AF 2-1D	MC, $M_{23}C_6$, M_6C
B-1900	MC, M_6C, M_3B_2.

The change of the amount of these constituents was recorded in graphical form such as that shown in Figure 5 for Alloy B-1900. (Note: The constituents were found as a result of aging at temperatures from 1400 to 2200 F but the temperature at which the phases were stable varied for each constituent and alloy).

Sigma in Hastelloy X

Aerojet-General has reported progress in its evaluation of Hastelloy X as a nuclear cladding.[6] Additional studies were undertaken to characterize the morphology of sigma formation in Hastelloy X after exposure to temperatures of 1300 and 1450 F. Residue samples obtained electrolytically from specimens containing sigma were combined and treated chemically with Murakami's reagent, which would selectively attack the carbide phases while leaving sigma phase relatively unaffected. X-ray diffraction analysis and photomicrography indicated that grain-boundary precipitates and regular-shaped

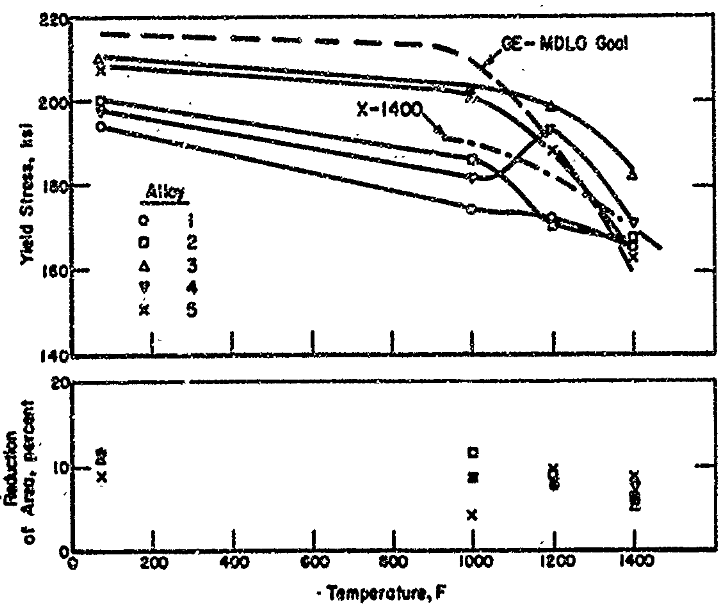

FIGURE 3. 0.2 PERCENT YIELD STRENGTH AND TENSILE DUCTILITY OF EXPERIMENTAL ALLOYS(4)

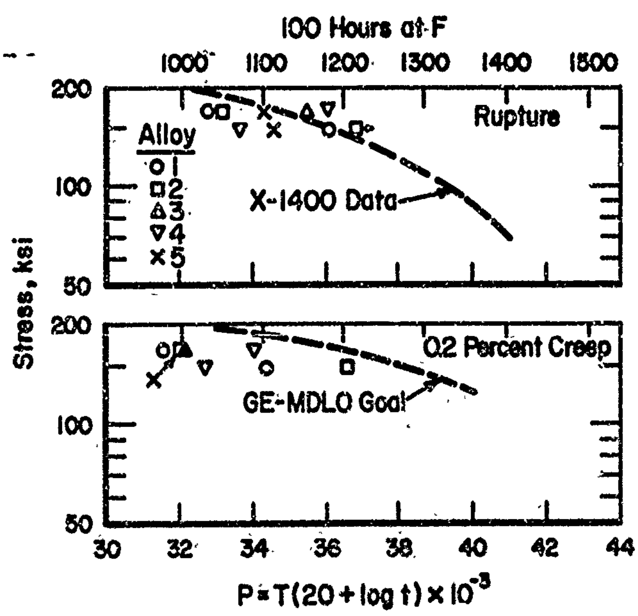

FIGURE 4. CREEP-RUPTURE DATA COMPARED WITH X-1400 DATA AND GE-MDLO GOAL(4)

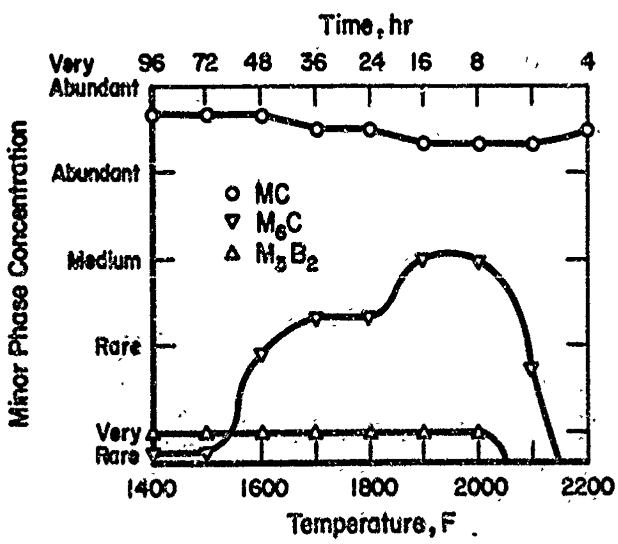

FIGURE 5. MINOR PHASE CONCENTRATION FOR B-1900 AS A FUNCTION OF AGING TEMPERATURE(5)

particles distributed throughout the matrix are M_6C carbide. The rod-shaped formations are sigma-phase. As pointed out, the standard pattern reported by ASTM for M_6C also lists the indices attributed to the sigma phase. Accordingly, it was recommended that the following d-spacings be used to identify sigma phase: 1.955, 1.995, 2.15, and 1.91. The d-spacings for positive identification of M_6C in Hastelloy X are as follows: 2.09, 2.22, 1.92, and 1.28.

Room-temperature tensile tests for tubing specimens aged for 7500 hours in air at 1900 and 2000 F and for sheet specimens aged at 1900 F were obtained. The results of tests of the 1900 F sheet specimens are plotted in Figure 6. The ultimate tensile strengths generally were found to decrease with exposure, but the elongation was not greatly changed. The results for tubing were similar.

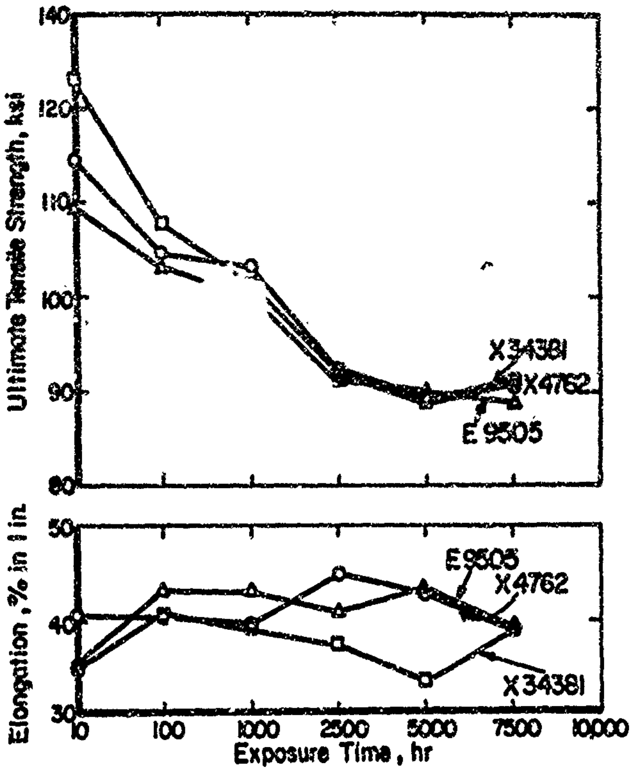

FIGURE 6. ROOM-TEMPERATURE TENSILE PROPERTIES OF VARIOUS HASTELLOY X SHEET HEATS EXPOSED IN AIR AT 1900 F(6)

PROCESS DEVELOPMENT

Union Carbide has reported on its program to roll Udimet 700 sheet.(7) Maximum hot ductility was obtained by overaging the material at 2050 F for 4 hours. Fabrication studies demonstrated that consumable-electrode vacuum-melted and vacuum-induction-melted material could be hot worked much better if canned than if bare. However, electroslag remelted material could be forged in the bare condition. Hot-rolling experiments on the latter material are not complete. Powder-consolidation techniques were attempted for the production of sheet, but were discontinued because of unsatisfactory results. Mechanical-property data on the sheet thus far produced show that at 1400 F, tensile, creep-rupture, and creep data are below those reported for bar. At 1800 F, the properties are better, but still do not compare with published bar data. Additional tests are under way to characterize further the properties of Udimet 700 sheet. It is believed that the loss of alloying elements at the surface of the sheet and during exposure to the test environment is responsible for the lower high-temperature properties. Future work will include evaluation of 15- and 20-mil-thick sheet. Also, the process will be scaled up, and approximately 400 pounds of sheet bar is to be processed into sheet. However, because the electroslag remelted material has not been completely evaluated, the choice of melting method is not certain at this time.

DISPERSION-STRENGTHENED ALLOYS

Nickel-Molybdenum Alloys

Teledyne Materials Research Company reported on its study of dispersion-strengthening nickel-molybdenum alloys.(8) The matrix was Ni-15Mo to which was added 2, 5, and 7.5 volume percent alumina or thoria. Selective reduction of oxides was chosen as the method of preparing the alloys. On the basis of the analysis of the structure before and after thermal-stability tests, three alloys were recommended for the evaluation of mechanical properties. These alloys contained 2 and 5 volume percent thoria, and 2 volume percent alumina. The tensile properties at room temperature and 2000 F are shown in Table 1. The creep-rupture properties at 2000 F in an argon atmosphere were determined, and it was found that the stresses for 3000-hour life were 600, 1000, and 450 psi, respectively, for the alloys containing 2 and 5 percent thoria and 2 percent alumina. These low values were attributed, at least in part, to the fact that the materials were tested in the as-extruded condition without any prior cold work. It was concluded, however, that very stable structures can be produced by the method of selective reduction of oxides.

Nickel-Molybdenum-Chromium Alloys

Melpar has reported on its attempt to produce a 70Ni-20Cr-10Mo dispersion-strengthened alloy by plating 0.1-micron-diameter zirconia particles successively with molybdenum, chromium, and nickel.(9) They found that vapor plating of fine oxide particles in a fluidized bed was suitable for producing nickel or chromium with apparently good oxide-particle distribution, although such samples are characterized by high carbon content. However, when attempts were made to vapor plate molybdenum by molybdenum pentachloride reduction at 575 C (1067 F), the zirconia particles agglomerated and sintered into 25 to 50-micron-diameter particles. It was concluded, therefore, that the fluidized-bed technique would be impossible to use to produce dispersion-strengthened alloys containing molybdenum unless suitable compounds were found that would decompose at lower temperatures and would not result in inherently high carbon content.

Utilization in Aerospace Structures

The alloy, Ni-20Cr-$2ThO_2$, has been selected by Douglas Aircraft Company for further development

TABLE 1. TENSILE PROPERTIES AT ROOM TEMPERATURE AND AT 2000 F OF EXTRUDED ALLOYS

Alloy	Dispersoid	Ultimate Tensile Strength, psi	0.02 Percent Yield Strength, psi	Elongation, percent	Reduction in Area, percent
		2000 F			
2	2 vol % ThO_2	6,430	4,950	7.2	5.9
3	2 vol % Al_2O_3	5,000	4,500	0.8	5.0
4	5 vol % ThO_2	5,440	4,450	--	18.8
5	5 vol % Al_2O_3	4,500	2,500	1.2	2.0
6	7.5 vol % ThO_2	5,100	3,000	8.6	8.1
		Room Temperature			
5	5 vol % Al_2O_3	131,100	115,300	2.4	3.1
6	7.5 vol% ThO_2	131,300	124,500	1.6	4.0

in Phase III of a program to develop structural design data, fabrication techniques, and a capability for structural utilization of dispersion-strengthened oxidation-resistant nickel-base and cobalt-base alloys.[10] In the joining tests, resistance spot welding, spot-diffusion bonding, brazing, and diffusion bonding have been investigated. Mechanical joining tests included the preparation of the sheet for flush rivets by dimpling and the performance of actual riveting tests with rivets of the same material. Fabrication experiments included shearing and slitting tests on 0.004-inch foil materials.

EVALUATION AND APPLICATIONS

Hypersonic Heat Shields

Solar Division of International Harvester Company has initiated a program on the evaluation of superalloys for hypersonic-vehicle honeycomb heat shields.[11] Six alloys selected for evaluation were Inconel 625, Haynes 25, René 41, Alloy 718, TDNi, and TDNiC. The effects of slow cyclic-oxidation exposure and long-time exposure stability will be investigated once base-line data have been obtained.

Udimet 630 for Turbine and Compressor Disks

Udimet 630 was examined for use as turbine and compressor disks in advanced engines.[12] It was reported that the material was notch sensitive when treated in the solution-treated (1950 F) and aged condition as recommended by the vendor. However, when compressor disk forgings were made at forging temperatures of 1860 and 1800 F, no tensile or rupture-notch sensitivity was observed when the forged material was solution heat treated at 1850 F followed by a standard age (1400 F, 10 hr, FC + 1200 F, 10 hr, AC). Material that had been "Mini-processed" gave superior tensile-fatigue and creep properties without notch sensitivity. Mini-processing is a proprietary thermomechanical treatment developed by Curtiss-Wright Corporation. Since the results of the investigation were from a single heat of material, further investigation was recommended before the material is adopted for use.

Welded Alloy 718

Stress-rupture data have been developed by Curtiss-Wright for welded Alloy 718 prepared by both inert-gas tungsten arc and the electron-beam processes.[13] The test established that both welding processes degrade the creep-rupture life of Alloy 718. The welding process caused a sensitized region to develop in the fusion line. The creep-rupture test at 1300 F caused a stress-induced boundary precipitation that exerted a detrimental influence on creep-rupture life. The specification called for a 24-hour life at 1300 F under a load of 70 ksi. Many of the electron-beam-welded specimens and TIG-welded specimens fell below this range. Almost all of the failures in the electron-beam-welded specimens were in the fusion line.

Aerojet-General, on the other hand, reported results of mechanical-property testing and welding of Alloy 718 for M-1 engine components.[14] The tests showed that Alloy 718 could be welded by electron-beam and TIG methods with high joint efficiencies. For example, in tests conducted at -320 F, ambient temperature, 600 F, 1200 F, and 1350 F, 100 percent joint efficiency was realized in all tests at all temperatures in solution-treated and aged weldments. In TIG-welded material, the weld joint efficiency, using tensile strength as the criterion, was approximately 93 percent at -423 F, and 95 percent at ambient temperature and 1200 F.

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